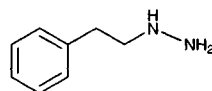


chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fen-camfamine, fenpropofen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiaicol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, imino-stilbene, imipramine, indomethacin, isocarboxtyril, isocarboxazid, isoniazid, isoproterenol, isox-suprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, mepredilone, mephentermine, mephenytoin, mephesis, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, meth-apyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyl dopa, methyl dopamine, methylphenidate, methylprednisolone, methyl-testosterone, methypyrrol, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, ox-ymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phenazocine, phenazopyridine, phenelzine, phenira-mine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenyl-ephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrila-mine, pyridylidone, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sufadiazine, sulfadimethoxine, sulfaethidole, sulfa-merazine, sulfamethazine, sulfamethoxazole, sulfanilamide, sulfapyridine, sulfasoxazole, sulin-dac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, the-obromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranlycy-promine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripeleminamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

## REFERENCE

Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233–242.

# Phenelzine



**Molecular formula:**  $C_8H_{12}N_2$

**Molecular weight:** 136.20

**CAS Registry No.:** 51-71-8, 156-51-4 (sulfate)

**Merck Index:** 7366

**Lednicer No.:** 1 74

## SAMPLE

**Matrix:** blood

**Sample preparation:** 2 mL Plasma + 400  $\mu$ L 10% acetic acid + 7 mL diethyl ether:dichloro-methane 2:1, shake, centrifuge at 2059 g for 10 min. Remove the aqueous layer and add it to 600  $\mu$ L 10% acetic acid, add 300  $\mu$ L 0.1% salicaldehyde in EtOH, heat at 60° for 30 min, cool to room temperature, add 1 mL 1 M  $K_2PO_4$  (sic), extract with 7 mL diethyl ether, centrifuge at 2059 g for 10 min, repeat extraction. Combine the organic layers and evaporate them to dryness under a stream of nitrogen at 40°, reconstitute the residue in 200  $\mu$ L buffer, inject a 20  $\mu$ L aliquot. (Buffer was 5 mM heptanesulfonic acid in MeCN:water:triethylamine 70:30:0.4.)

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**HPLC VARIABLES****Guard column:** 50 × 4.6 30 μm C8**Column:** 250 × 4.6 Spherisorb S5 ODS2 C18**Mobile phase:** Gradient. MeCN:buffer:water 0:75:25 for 5 min, 15:85:0 for 12 min (step gradient).  
(Buffer was 5 mM heptanesulfonic acid in MeCN:water:triethylamine 70:30:0.4.)**Flow rate:** 1**Injection volume:** 20**Detector:** UV 280

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**CHROMATOGRAM****Retention time:** 11.09**Internal standard:** phenelzine

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**OTHER SUBSTANCES****Extracted:** isoniazid, hydrazine, monoacetylhydrazine

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**KEY WORDS**derivatization; phenelzine is IS

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**REFERENCE**Walubo,A.; Smith,P.; Folb,P.I. Comprehensive assay for pyrazinamide, rifampicin and isoniazid with its hydrazine metabolites in human plasma by column liquid chromatography, *J.Chromatogr.B*, **1994**, 658, 391–396.

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**SAMPLE****Matrix:** blood, CSF**Sample preparation:** 200-500 μL Plasma or CSF + 100 μL 10% (?) aqueous acetic acid + 5 mL n-hexane, shake for 30 min, centrifuge at 1870 g for 10 min. Discard the organic layer. Add 300 μL 0.1% salicaldehyde in EtOH and 400 μL 10% aqueous acetic acid to the aqueous layer, heat at 60° for 30 min, cool, add 1 mL 1 M pH 6.5 K<sub>2</sub>HPO<sub>4</sub>, shake for 10 s, add 5 mL diethyl ether, shake for 10 min, centrifuge at 1870 g for 10 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue in 50 μL mobile phase, inject a 25 μL aliquot.

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**HPLC VARIABLES****Guard column:** 30 × 4.6 30 μm C8 (Waters)**Column:** 300 × 3.9 10 μm μBondapak C18**Mobile phase:** MeCN:water:triethylamine 70:30:0.4 containing 5 mM heptanesulfonic acid, pH adjusted to 6.0 with acetic acid**Flow rate:** 1**Injection volume:** 25**Detector:** UV 320

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**CHROMATOGRAM****Retention time:** 3**Internal standard:** phenelzine sulfate

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**OTHER SUBSTANCES****Extracted:** hydrazine, isoniazid**Noninterfering:** p-aminosalicylic acid, pyrazinamide, rifampin

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**KEY WORDS**plasma; rabbit; derivatization; phenelzine is IS

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**REFERENCE**Walubo,A.; Chan,K.; Wong,C.L. Simultaneous assay for isoniazid and hydrazine metabolite in plasma and cerebrospinal fluid in the rabbit, *J.Chromatogr.*, **1991**, 567, 261–266.

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**SAMPLE****Matrix:** formulations**Sample preparation:** Mix powdered tablet with 5 mL 200 mM pH 6 sodium acetate buffer, rotate at 30 rpm for 30 min, centrifuge. Remove a 1 mL aliquot of the supernatant and add it

to 1 mL 15 mg/mL benzaldehyde in MeOH:water 50:50, rotate at 30 rpm for 10 min, add 20 mL 3 µg/mL IS in mobile phase, rotate at 30 rpm for 30 min, inject a 50 µL aliquot of the upper layer.

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**HPLC VARIABLES**

**Column:** 150 × 4.6 Ultrasphere Si

**Mobile phase:** n-Hexane:chloroform 95:5

**Flow rate:** 2

**Injection volume:** 50

**Detector:** UV 313

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**CHROMATOGRAM**

**Retention time:** 17

**Internal standard:** iminodibenzyl (2)

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**OTHER SUBSTANCES**

**Simultaneous:** hydrazine

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**KEY WORDS**

derivatization; tablets; normal phase

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**REFERENCE**

Matsui,F.F.; Butterfield,A.G.; Curran,N.M.; Lovering,E.G.; Sears,R.W.; Robertson,D.L. Determination of hydrazine in pharmaceuticals. Part 2. Phenelzine sulfate, *Can.J.Pharm.Sci.*, **1981**, 16, 20–22.

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**SAMPLE**

**Matrix:** solutions

**Sample preparation:** Dissolve in MeOH at a concentration of 1 mg/mL, inject a 20 µL aliquot.

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**HPLC VARIABLES**

**Column:** 250 × 5 Spherisorb S5W

**Mobile phase:** MeOH:buffer 90:10 (Buffer was 94 mL 35% ammonia and 21.5 mL 70% nitric acid in 884 mL water, adjust the pH to 10.1 with ammonia.)

**Flow rate:** 2

**Injection volume:** 20

**Detector:** UV 254

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**CHROMATOGRAM**

**Retention time:** 1.81

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**OTHER SUBSTANCES**

**Simultaneous:** normethadone, meperidine, dipipanone, diamorphine, pentazocine, acetylcodeine, monoacetylmorphine, thebacon, oxycodone, thebaine, norlevorphanol, methadone, benzylmorphine, ethylmorphine, morphine-N-oxide, codeine, codeine-N-oxide, morphine, ethoheptazine, morphine-3-glucuronide, pholcodeine, norpethidine, hydrocodone, dihydrocodeine, dihydromorphine, levorphanol, norcodeine, normorphine, pemoline, benzphetamine, diethylpropion, mazindol, epinephrine, pipradol, phenylpropanolamine, fencamfamin, chlorphentermine, norpseudoephedrine, phentermine, fenfluramine, methylenedioxymphetamine, amphetamine, normetanephine, 4-hydroxyamphetamine, bromo-STP, STP, prolintane, 2-phenethylamine, tyramine, trimethoxyamphetamine, phenylephrine, pseudoephedrine, ephedrine, methylephedrine, dimethylamphetamine, methamphetamine, mescaline, mephentermine, buprenorphine, dextromoramide, phenoperidine, fentanyl, etorphine, piritramide, noscapine, papaverine, naloxone, dextropropoxyphene

**Noninterfering:** dopamine, levodopa, methyl dopa, methyl dopate, norepinephrine

**Interfering:** tranlylcypromine, caffeine, fenethyline, phendimetrazine, methylphenidate, nalorphine, phenazocine, norpipanone, levallorphan, hydroxyphenidine

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**REFERENCE**

Law,B.; Gill,R.; Moffat,A.C. High-performance liquid chromatography retention data for 84 basic drugs of forensic interest on a silica column using an aqueous methanol eluent, *J.Chromatogr.*, **1984**, 301, 165–172.

**SAMPLE****Matrix:** solutions**Sample preparation:** Prepare a 10 µg/mL solution in MeOH, inject a 20 µL aliquot.**HPLC VARIABLES****Column:** 125 × 4.9 Spherisorb S5W silica**Mobile phase:** MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7**Flow rate:** 2**Injection volume:** 20**Detector:** E, LeCarbone, V25 glassy carbon electrode, + 1.2 V**CHROMATOGRAM****Retention time:** 1.8**OTHER SUBSTANCES**

**Also analyzed:** acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipipanonone, diprenorphine, dipyridamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethiopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserine, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamlamine, meclorphenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylegonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, piminodine, pimozone, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thiopropazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranlycypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripeleminamine, triprolidine, tryptamine, verapamil, xylometazoline

**REFERENCE**

Jane, L.; McKinnon, A.; Flanagan, R.J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J. Chromatogr.*, **1985**, *323*, 191–225.

**SAMPLE****Matrix:** solutions**HPLC VARIABLES****Column:** 250 × 4.6 Zorbax RX

**Mobile phase:** Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.

**Column temperature:** 30

**Flow rate:** 2

**Detector:** UV 210

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## OTHER SUBSTANCES

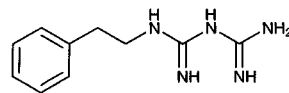
**Also analyzed:** acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitrityline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepam, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlor-diazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fencamfamine, fenopropfen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiaicol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, iminostilbene, imipramine, indomethacin, isocarboxtyril, isocarboxazid, isoniazid, isoproterenol, isoxsuprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, meperidine, mephentermine, mephenytoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, methapyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyl dopa, methyl dopamine, methylphenidate, methylprednisolone, methyltestosterone, methyprylon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nyldrin, oxazepam, oxycodone, oxymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phenazocine, phenazopyridine, phencyclidine, pheniramine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenylephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrillamine, pyrrithyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sufadiazine, sulfadimethoxine, sulfaethidole, sulfamerazine, sulfamethazine, sulfamethoxazole, sulfanilamide, sulfapyridine, sulfasoxazole, sulindac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, theobromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranlycypromine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripeleennamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

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## REFERENCE

Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233-242.

# Phenformin



**Molecular formula:**  $C_{10}H_{15}N_5$

**Molecular weight:** 205.26

**CAS Registry No.:** 114-86-3, 834-28-6 (HCl)

**Merck Index:** 7376

**Lednicer No.:** 1 75

## SAMPLE

**Matrix:** microsomal incubations

**Sample preparation:** Mix 2 mL microsomal incubation with 4 mL MeOH:DMSO 80:20, centrifuge at 300 g for 20 min, dilute 1:2 with water, inject an aliquot.

## HPLC VARIABLES

**Column:** 250 × 4.6 5  $\mu$ m Hypersil-BDS C18

**Mobile phase:** MeOH:100 mM pH 6.5 ammonium acetate 20:80

**Flow rate:** 1

**Injection volume:** 200-500

**Detector:** UV 236; MS, VG Quattro BQ tandem quadrupole, API, ESI, positive ion mode, source 150°, cone voltage and lens 2 40 and 50 V, m/z 206

## CHROMATOGRAM

**Retention time:** 19

## OTHER SUBSTANCES

**Extracted:** metabolites

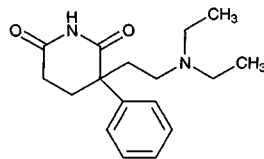
## KEY WORDS

comparison with capillary electrophoresis; rat; liver

## REFERENCE

Llambias, E.B.; Luo, J. Study of phenformin metabolism in rat liver microsomes by HPLC, CE and on-line HPLC-electrospray ionization mass spectrometry, *Biomed. Chromatogr.*, **1996**, 10, 155-160.

# Phenglutarimide



**Molecular formula:**  $C_{17}H_{24}N_2O_2$

**Molecular weight:** 288.39

**CAS Registry No.:** 1156-05-4, 1674-96-0 (HCl)

**Merck Index:** 7377

## SAMPLE

**Matrix:** solutions

**Sample preparation:** Prepare a 10  $\mu$ g/mL solution in MeOH, inject a 20  $\mu$ L aliquot.

## HPLC VARIABLES

**Column:** 125 × 4.9 Spherisorb S5W silica

**Mobile phase:** MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7

**Flow rate:** 2

**Injection volume:** 20

**Detector:** E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

## CHROMATOGRAM

**Retention time:** 3.6

## OTHER SUBSTANCES

**Also analyzed:** acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzocetamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclopropoxyphene, dicyclomine, diethylcarbamazepine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipiprone, diprenorphine, dipyrindamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserine, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamylamine, meclorphenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylexgonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, pimindine, pimozone, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirazepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, propeptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thiopropazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranlycypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripeleminamine, triprolidine, tryptamine, verapamil, xylometazoline

## REFERENCE

Jane, I.; McKinnon, A.; Flanagan, R. J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J. Chromatogr.*, **1985**, 323, 191-225.

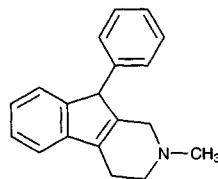
# Phenindamine

**Molecular formula:** C<sub>19</sub>H<sub>19</sub>N

**Molecular weight:** 261.37

**CAS Registry No.:** 82-88-2, 569-59-5 (tartrate)

**Merck Index:** 7380



## SAMPLE

**Matrix:** bulk

**Sample preparation:** Disperse 100 mg phenindamine tartrate in 5 mL 1 mM nitric acid with gentle shaking, make up to 10 mL with MeOH, shake until dissolution is complete, inject a 5 µL aliquot.

## HPLC VARIABLES

**Column:** µBondapak C18

**Mobile phase:** MeOH:buffer 50:50 (Protect mobile phase from light, prepare fresh daily.) (Buffer was 40 g/L silver nitrate in 1 mM nitric acid.)

**Flow rate:** 0.8  
**Injection volume:** 5  
**Detector:** UV 254

**CHROMATOGRAM**

**Retention time:** 15

**OTHER SUBSTANCES**

**Simultaneous:** isophenindamine

**KEY WORDS**

protect from light

**REFERENCE**

Tscherne, R.J.; Umagat, H. Determination of isophenindamine in phenindamine tartrate using an argentated high-performance liquid chromatographic mobile phase, *J. Pharm. Sci.*, **1980**, 69, 342-344.

**SAMPLE**

**Matrix:** solutions

**Sample preparation:** Prepare a 10 µg/mL solution in MeOH, inject a 20 µL aliquot.

**HPLC VARIABLES**

**Column:** 125 × 4.9 Spherisorb S5W silica

**Mobile phase:** MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7

**Flow rate:** 2

**Injection volume:** 20

**Detector:** E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

**CHROMATOGRAM**

**Retention time:** 3.2

**OTHER SUBSTANCES**

**Also analyzed:** acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazepine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipiprone, diprenorphine, dipyrindamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserin, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamlamine, meclorphenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypropazine, methylephedrine, methylergonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, piminozide, pimozide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine,



prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranlycypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripeleppamine, triprolidine, tryptamine, verapamil, xylometazoline

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**REFERENCE**

Jane, I.; McKinnon, A.; Flanagan, R. J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J. Chromatogr.*, **1985**, 323, 191–225.

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**SAMPLE**

**Matrix:** solutions

**Sample preparation:** Dissolve in MeOH:water 1:1 at a concentration of 50 µg/mL, inject a 10 µL aliquot.

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**HPLC VARIABLES**

**Column:** 300 × 3.9 10 µm µBondapak C18

**Mobile phase:** MeOH:acetic acid:triethylamine:water 50:1.5:0.5:48

**Flow rate:** 1.5

**Injection volume:** 10

**Detector:** UV 254

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**CHROMATOGRAM**

**Retention time:** 10

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**OTHER SUBSTANCES**

**Simultaneous:** thonzylamine, pheniramine, tripeleppamine, chlorpheniramine, brompheniramine, phenyltoxamine, clemizole

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**REFERENCE**

Roos, R. W.; Lau-Cam, C. A. General reversed-phase high-performance liquid chromatographic method for the separation of drugs using triethylamine as a competing base, *J. Chromatogr.*, **1986**, 370, 403–418.

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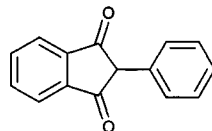
# Phenindione

**Molecular formula:** C<sub>15</sub>H<sub>10</sub>O<sub>2</sub>

**Molecular weight:** 222.24

**CAS Registry No.:** 83-12-5

**Merck Index:** 7381



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**SAMPLE**

**Matrix:** blood, urine

**Sample preparation:** Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 µL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) µL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200–350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

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**HPLC VARIABLES**

**Guard column:** 20 mm long Symmetry C18

**Column:** 250 × 4.6 5 µm Symmetry C8 (Waters)

**Mobile phase:** Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85:15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.

**Column temperature:** 30

**Flow rate:** 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)

**Injection volume:** 10-30

**Detector:** UV 226.3

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## CHROMATOGRAM

**Retention time:** 18.062

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## KEY WORDS

whole blood

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## REFERENCE

Gaillard,X.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, 763, 149-163.

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# Pheniramine

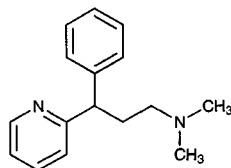
**Molecular formula:** C<sub>16</sub>H<sub>20</sub>N<sub>2</sub>

**Molecular weight:** 240.35

**CAS Registry No.:** 86-21-5, 132-20-7 (maleate)

**Merck Index:** 7383

**Lednicer No.:** 1 77



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## SAMPLE

**Matrix:** blood

**Sample preparation:** 1 mL Plasma + 30 µL 10 µg/mL amitriptyline in water + 500 µL 1 M sodium carbonate, vortex for 30 s, add 5 mL diethyl ether, vortex for 2 min, centrifuge at 4000 rpm for 10 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue in 250 µL mobile phase, vortex for 30 s, centrifuge at 12000 rpm for 5 min, inject an aliquot of the supernatant.

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## HPLC VARIABLES

**Column:** 300 × 3.9 10 µm µBondapak C18

**Mobile phase:** MeOH:water 62:38 adjusted to pH 3.5 with phosphoric acid

**Column temperature:** 40

**Flow rate:** 1.2

**Detector:** UV 262

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## CHROMATOGRAM

**Retention time:** 4.5

**Internal standard:** amitriptyline (6.1)

**Limit of detection:** 10 ng/mL

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## OTHER SUBSTANCES

**Simultaneous:** diazepam, diltiazem, flurbiprofen, ibuprofen, itraconazole, ketoprofen, mebeverine, metoclopramide, phenylbutazone

**Interfering:** chlorpheniramine

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## KEY WORDS

plasma; pharmacokinetics; dog

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**REFERENCE**

El-Sayed, Y.M.; Niazy, E.M.; Khidir, S.H. High-performance liquid chromatographic method for the quantitative determination of pheniramine in plasma, *J. Liq. Chromatogr.*, **1995**, *18*, 763–777.

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**SAMPLE**

**Matrix:** blood, tissue

**Sample preparation:** Blood or serum. 1 mL Blood or serum + 1 µg cianopramine + 1 mL water, vortex, add 1 mL 200 mM sodium carbonate, vortex, add 6 mL hexane:1-butanol 95:5, gently agitate for 30 min, centrifuge at 2500 g for 5 min. Remove the organic layer and add it to 100 µL 0.2% phosphoric acid, agitate gently for 30 min, centrifuge for 5 min. Remove the organic layer and inject a 30 µL aliquot of the aqueous layer. Liver homogenate. 0.5 mL Liver homogenate + 10 µg cianopramine + 500 µL 2% sodium tetraborate + 8 mL hexane:1-butanol 95:5, gently agitate for 30 min, centrifuge at 2500 g for 5 min. Remove the organic layer and add it to 400 µL 0.2% phosphoric acid, agitate gently for 30 min, centrifuge for 5 min. Remove the organic layer and inject a 30 µL aliquot of the aqueous layer.

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**HPLC VARIABLES**

**Guard column:** 15 × 3.2 7 µm RP-18 Newguard (Applied Biosystems)

**Column:** 100 × 4.6 5 µm Brownlee Spheri-5 RP-18

**Mobile phase:** MeCN:100 mM NaH<sub>2</sub>PO<sub>4</sub>:diethylamine 40:57.5:2.5

**Flow rate:** 2

**Injection volume:** 30

**Detector:** UV 220

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**CHROMATOGRAM**

**Retention time:** 4.07

**Internal standard:** cianopramine (8.93)

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**OTHER SUBSTANCES**

**Simultaneous:** amitriptyline, amoxapine, benztropine, brompheniramine, chlorpheniramine, chlorpromazine, clomipramine, cyproheptadine, desipramine, diphenhydramine, dothiepin, doxepin, fluoxetine, haloperidol, imipramine, loxapine, maprotiline, meperidine, mesoridazine, methadone, metoclopramide, mianserin, moclobemide, nomifensine, nordoxepin, norfluoxetine, norpropoxyphene, nortriaden, nortriptyline, pentobarbital, promethazine, propoxyphene, propranolol, protriptyline, quinidine, quinine, sulforidazine, thioridazine, thiothixene, tranlycypromine, trazodone, trihexyphenidyl, trimipramine, triprolidine

**Noninterfering:** dextromethorphan, norphetidine, phenoxybenzamine, prochlorperazine, trifluoperazine

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**KEY WORDS**

serum; whole blood; liver

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**REFERENCE**

McIntyre, I.M.; King, C.V.; Skafidis, S.; Drummer, O.H. Dual ultraviolet wavelength high-performance liquid chromatographic method for the forensic or clinical analysis of seventeen antidepressants and some selected metabolites, *J. Chromatogr.*, **1993**, *621*, 215–223.

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**SAMPLE**

**Matrix:** cell incubations

**Sample preparation:** 40 mL Cell incubation + 50 mL MeOH, shake vigorously for 1 min, centrifuge at 2000 rpm for 10 min, wash the pellet twice with 50 mL portions of MeOH. Combine the supernatants and add 100 mL water, extract three times with 150 mL portions of dichloromethane. Filter the extracts through anhydrous sodium sulfate, evaporate the filtrate to dryness under reduced pressure at 40°, reconstitute with mobile phase, inject an aliquot.

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**HPLC VARIABLES**

**Column:** 250 × 4.6 5 µm cyano-propyl (Beckman)

**Mobile phase:** MeCN:buffer 40:60 (Buffer was 10 mM KH<sub>2</sub>PO<sub>4</sub> containing 20 mM triethylamine, pH 7.0.)

**Flow rate:** 1

**Injection volume:** 20

**Detector:** UV 254

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**CHROMATOGRAM****Retention time:** 8.9

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**OTHER SUBSTANCES****Extracted:** metabolites**Interfering:** brompheniramine, chlorpheniramine

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**KEY WORDS**

also semipreparative details

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**REFERENCE**

Hansen, E.B., Jr.; Cho, B.P.; Korfmacher, W.A.; Cerniglia, C.E. Fungal transformations of antihistamines: metabolism of brompheniramine, chlorpheniramine, and pheniramine to *N*-oxide and *N*-demethylated metabolites by the fungus *Cunninghamella elegans*, *Xenobiotica*, **1995**, 25, 1081–1092.

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**SAMPLE****Matrix:** formulations

**Sample preparation:** Crush 10 tablets, add 250 mL 50 mM HCl in EtOH:water 50:50, heat for 15 min on a steam bath, shake mechanically for 2 h, filter (glass fiber GF/A, Whatman), inject a 30  $\mu$ L aliquot of the filtrate.

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**HPLC VARIABLES****Column:** 250  $\times$  4.6 10  $\mu$ m Partisil-10-ODS

**Mobile phase:** MeCN:buffer 50:50 (Buffer was 2.85 mM ethylenediamine sulfate adjusted to pH 7.44  $\pm$  0.02 with 1 M ammonium hydroxide.)

**Flow rate:** 3.8**Injection volume:** 30**Detector:** UV 216.5

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**CHROMATOGRAM****Retention time:** 20

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**OTHER SUBSTANCES****Simultaneous:** aposcopolamine, methscopolamine, phenylpropanolamine, pyrilamine, tropic acid

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**KEY WORDS**

tablets

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**REFERENCE**

Heidemann, D.R. High-pressure liquid chromatographic determination of methscopolamine nitrate, phenylpropanolamine hydrochloride, pyrilamine maleate, and pheniramine maleate in tablets, *J.Pharm.Sci.*, **1981**, 70, 820–822.

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**SAMPLE****Matrix:** formulations

**Sample preparation:** Tablets. One tablet + 50 mL MeOH, sonicate, make up to 100 mL with MeOH, centrifuge for 15 min. Remove 1 mL supernatant, make up to 10 mL with mobile phase, inject a 50  $\mu$ L aliquot. Drops. Dilute drops with the mobile phase so that the concentration of pheniramine maleate is 25  $\mu$ g/mL, inject a 50  $\mu$ L aliquot.

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**HPLC VARIABLES****Column:** 100  $\times$  4.6 Cyclobond I (Advanced Separation Technologies)

**Mobile phase:** MeOH:50 mM  $\text{NaH}_2\text{PO}_4$  adjusted to pH 7.0 with 0.1 M NaOH 30:70

**Column temperature:** 35**Flow rate:** 1.5**Injection volume:** 50**Detector:** UV 254

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**CHROMATOGRAM****Retention time:** 6.2

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**OTHER SUBSTANCES**

**Simultaneous:** pyrilamine (mepyramine), phenylpropanolamine

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**KEY WORDS**

tablets; drops

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**REFERENCE**

el-Gizawy,S.M.; Ahmed,A. High-performance liquid chromatographic determination of mepyramine maleate, pheniramine maleate and phenylpropanolamine hydrochloride in tablets and drops, *Analyst*, **1987**, *112*, 867-869.

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**SAMPLE**

**Matrix:** formulations, urine

**Sample preparation:** Tablets. Crush tablets, add 100 mL water and 30-40 mL MeCN, dissolve, add N,N-dimethylbenzylamine, make up to 250 or 500 mL with water, centrifuge an aliquot, inject a 20  $\mu$ L aliquot of the supernatant. Urine. Inject a 100  $\mu$ L aliquot of urine directly.

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**HPLC VARIABLES**

**Column:** 150  $\times$  4.6 Asahipak ODP-50 C18

**Mobile phase:** MeCN:200 mM pH 7.0 phosphate buffer 27:73

**Flow rate:** 0.8

**Injection volume:** 20-100

**Detector:** Chemiluminescence following post-column reaction. Oxidize a 1 mM tris(2,2'-bipyridine) ruthenium(II) hexachloride solution in 50 mM pH 5.5 acetate buffer to Ru(III) using a Princeton Applied Research polarographic analyzer with a platinum gauze working electrode, platinum wire auxiliary electrode, and a silver wire reference electrode, +950 mV. Pump the reagent solution at 0.28 mL/min and mix with the column effluent, allow to flow through detector. The chemiluminescence detector was a fluorescence detector with the light source removed.

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**CHROMATOGRAM**

**Retention time:** 3.5

**Internal standard:** N,N-dimethylbenzylamine

**Limit of detection:** 90 ng/mL

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**OTHER SUBSTANCES**

**Simultaneous:** brompheniramine, chlorpheniramine, pyrilamine, diphenhydramine

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**KEY WORDS**

tablets

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**REFERENCE**

Holeman,J.A.; Danielson,N.D. Liquid chromatography of antihistamines using post-column tris(2, 2'-bipyridine) ruthenium(III) chemiluminescence detection, *J.Chromatogr.A*, **1994**, *679*, 277-284.

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**SAMPLE**

**Matrix:** solutions

**Sample preparation:** Prepare a 10  $\mu$ g/mL solution in MeOH, inject a 20  $\mu$ L aliquot.

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**HPLC VARIABLES**

**Column:** 125  $\times$  4.9 Spherisorb S5W silica

**Mobile phase:** MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7

**Flow rate:** 2

**Injection volume:** 20

**Detector:** E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

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**CHROMATOGRAM**

**Retention time:** 4.5

**OTHER SUBSTANCES**

**Also analyzed:** acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipiprone, diprenorphine, dipyrindamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothiopyndyl, isoxsuprine, ketanserine, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamlamine, meclophenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylegonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, piminodine, pimozone, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranlycypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripeleminamine, triprolidine, tryptamine, verapamil, xylometazoline

**REFERENCE**

Jane, I.; McKinnon, A.; Flanagan, R. J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J. Chromatogr.*, **1985**, *323*, 191–225.

**SAMPLE**

**Matrix:** solutions

**HPLC VARIABLES**

**Column:** 305 × 7 PRP-1 (Hamilton)

**Mobile phase:** Gradient. A was water:triethylamine 99.9:0.1. B was MeCN:triethylamine 99.9:0.1. A:B 60:40 for 7 min, to 20:80 over 5 min, maintain at 20:80 for 5 min, to 60:40 over 6 min, re-equilibrate at 60:40 for 2 min.

**Column temperature:** 40

**Flow rate:** 3.5

**Injection volume:** 500

**Detector:** UV 254

**CHROMATOGRAM**

**Retention time:** 12.0

**OTHER SUBSTANCES**

**Simultaneous:** diphenylpyraline, doxylamine, guaifenesin, hydrocodone, phenylephrine, phenylpropanolamine, pyrilamine

**Interfering:** etafedrine

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**REFERENCE**

Black,D.B.; By,A.W.; Lodge,B.A. Isolation and identification of hydrocodone in narcotic cough syrups by high-performance liquid chromatography with infrared spectrometric identification, *J.Chromatogr.*, **1986**, 358, 438-443.

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**SAMPLE**

**Matrix:** solutions

**Sample preparation:** Dissolve in MeOH:water 1:1 at a concentration of 50 µg/mL, inject a 10 µL aliquot.

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**HPLC VARIABLES**

**Column:** 300 × 3.9 10 µm µBondapak C18

**Mobile phase:** MeOH:acetic acid:triethylamine:water 50:1.5:0.5:48

**Flow rate:** 1.5

**Injection volume:** 10

**Detector:** UV 254

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**CHROMATOGRAM**

**Retention time:** 4

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**OTHER SUBSTANCES**

**Simultaneous:** thonzylamine, tripeleennamine, chlorpheniramine, brompheniramine, phenindamine, phenyltoxamine, clemizole

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**REFERENCE**

Roos,R.W.; Lau-Cam,C.A. General reversed-phase high-performance liquid chromatographic method for the separation of drugs using triethylamine as a competing base, *J.Chromatogr.*, **1986**, 370, 403-418.

---

**SAMPLE**

**Matrix:** solutions

**Sample preparation:** Inject a 50 µL aliquot.

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**HPLC VARIABLES**

**Column:** 300 × 4 µBondapak phenyl

**Mobile phase:** MeCN:0.01% phosphoric acid containing 0.01% NaCl 35:65, final pH 2.8

**Flow rate:** 1.5

**Injection volume:** 50

**Detector:** UV 210

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**CHROMATOGRAM**

**Retention time:** 3.4

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**OTHER SUBSTANCES**

**Simultaneous:** amitriptyline, buprenorphine, chlorpromazine, cocaine, desipramine, desmethyldoxepin, dextromoramide, diphenhydramine, doxepin, imipramine, meperidine, methadone, normeperidine, norpropoxyphene, nortriptyline, oxazepam, pentazocine, pericyazine, propoxyphene, propranolol, quinine, thiopropazate, thioridazine

**Interfering:** codeine, ephedrine, oxycodone

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**REFERENCE**

Hackett,L.P.; Dusci,L.J.; Ilett,K.F. The analysis of several nonopiate narcotic analgesics and cocaine in serum using high-performance liquid chromatography, *J.Anal.Toxicol.*, **1987**, 11, 269-271.

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**SAMPLE**

**Matrix:** solutions

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**HPLC VARIABLES**

**Column:** 250 × 4.6 5 µm Ultrasphere cyano

**Mobile phase:** MeCN:10 mM pH 2.5 KH<sub>2</sub>PO<sub>4</sub> 60:40

**Flow rate:** 2.5

**Injection volume:** 20-40

**Detector:** E, Environmental Science Associates Coulochem Model 5100A, Model 5100 guard cell +0.85 V (between pump and injector), Model 5010 analytical cell +0.8 V, pre-analytical cell +0.3 V

## CHROMATOGRAM

**Retention time:** 4.9

## OTHER SUBSTANCES

**Simultaneous:** amitriptyline, amoxapine, chlorpromazine, desmethyldoxepin, fluphenazine, mesoridazine, perphenazine, phenylephrine, prochlorperazine, reduced haloperidol, thioridazine, thiothixene, trazodone, triflupromazine, trimetopazine, tripeleminamine

**Noninterfering:** diazepam, diphenhydramine, ethopropazine, fluoxetine, nordiazepam, oxazepam, phenylpropanolamine, pseudoephedrine, trifluoperazine

**Interfering:** desipramine, doxepin, haloperidol, imipramine, loxapine, nortriptyline, promazine, promethazine

## REFERENCE

Hariharan,M.; VanNoord,T.; Kindt,E.K.; Tandon,R. A simple, sensitive liquid chromatographic assay of cis-thiothixene in plasma with coulometric detection, *Ther.Drug Monit.*, **1991**, *13*, 79-85.

## SAMPLE

**Matrix:** solutions

## HPLC VARIABLES

**Column:** 250 × 4.6 Zorbax RX

**Mobile phase:** Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.

**Column temperature:** 30

**Flow rate:** 2

**Detector:** UV 210

## OTHER SUBSTANCES

**Also analyzed:** acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitriptyline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepam, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlor-diazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fen-camfamine, fenpropofen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiaicol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, imino-stilbene, imipramine, indomethacin, isocarboxystyryl, isocarboxazid, isoniazid, isoproterenol, isox-suprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megesterol, mepacrine, meperidine, mephentermine, mephentyoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, meth-apyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methylodopa, methylodopamine, methylphenidate, methylprednisolone, methyltestosterone, methypyrrolon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, ox-



ymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phenazocine, phenazopyridine, phencyclidine, phendimetrazine, phenobarbital, phenothiazine, phenoxymizole, phentermine, phenylbutazone, phenylephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrilamine, pyrrhyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sulfadiazine, sulfadimethoxine, sulfaethidole, sulfamerazine, sulfamethazine, sulfamethoxazole, sulfanilamide, sulfapyridine, sulfasoxazole, sulindac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, theobromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranlycypromine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripeleminamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

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## REFERENCE

Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233-242.

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## SAMPLE

**Matrix:** solutions

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## HPLC VARIABLES

**Column:** 150 × 4.6 12 µm 1-myristoyl-2-[(13-carboxyl)-tridecoyl]-sn-3-glycerophosphocholine chemically bonded to silica (Regis)

**Mobile phase:** MeCN:100 mM pH 7.0 phosphate buffer 20:80

**Flow rate:** 1

**Detector:** UV 254

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## CHROMATOGRAM

**Retention time:** k' 4.00

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## OTHER SUBSTANCES

**Also analyzed:** acebutolol, alprenolol, antazoline, atenolol, betaxolol, bisoprolol, bopindolol, bupranolol, carteolol, celiprolol, chloropyramine, chlorpheniramine, cicloprolol, cimetidine, cinarizine, cirazoline, clonidine, dilevalol, dimethindene, diphenhydramine, doxazosin, esmolol, famotidine, isothipendyl, ketotifen, metiamide, metoprolol, moxonidine, nadolol, naphazoline, nifenalol, nizatidine, oxprenolol, phentolamine, pindolol, pizotyline (pizotifen), practolol, prazosin, promethazine, propranolol, pyrilamine (mepyramine), ranitidine, roxatidine, sotalol, tiamenidine, timolol, tramazoline, tripeleminamine, triprolidine, tymazoline, UK-14,304

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## REFERENCE

Kaliszan,R.; Nasal,A.; Turowski,M. Binding site for basic drugs on α<sub>1</sub>-acid glycoprotein as revealed by chemometric analysis of biochromatographic data, *Biomed.Chromatogr.*, **1995**, *9*, 211-215.

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## SAMPLE

**Matrix:** solutions

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## HPLC VARIABLES

**Column:** 250 × 4.6 5 µm Supelcosil LC-DP (A) or 250 × 4 5 µm LiChrospher 100 RP-8 (B)

**Mobile phase:** MeCN:0.025% phosphoric acid:buffer 25:10:5 (A) or 60:25:15 (B) (Buffer was 9 mL concentrated phosphoric acid and 10 mL triethylamine in 900 mL water, adjust pH to 3.4 with dilute phosphoric acid, make up to 1 L.)

**Flow rate:** 0.6

**Injection volume:** 25

**Detector:** UV 229

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## CHROMATOGRAM

**Retention time:** 9.49 (A), 4.49 (B)

**OTHER SUBSTANCES**

**Also analyzed:** acebutolol, acepromazine, acetaminophen, acetazolamide, acetophenazine, albuterol, alprazolam, amitriptyline, amobarbital, amoxapine, antipyrine, atenolol, atropine, azatadine, baclofen, benzocaine, bromocriptine, brompheniramine, brotizolam, bupivacaine, buspirone, butabarbital, butalbital, caffeine, carbamazepine, cetirizine, chlorcyclizine, chlordi-azepoxide, chlormezanone, chloroquine, chlorpheniramine, chlorpromazine, chlorpropamide, chlorprothixene, chlorthalidone, chlorzoxazone, cimetidine, cisapride, clomipramine, clonazepam, clonidine, clozapine, cocaine, codeine, colchicine, cyclizine, cyclobenzaprine, dantrolene, desipramine, diazepam, diclofenac, diflunisal, diltiazem, diphenhydramine, diphenidol, diphen-oxylate, dipyridamole, disopyramide, dobutamine, doxapram, doxepin, droperidol, encainide, ethidium bromide, ethopropazine, fenoprofen, fentanyl, flavoxate, fluoxetine, fluphenazine, flur-azepam, flurbiprofen, fluvoxamine, furosemide, glutethimide, glyburide, guaifenesin, haloper- idol, homatropine, hydralazine, hydrochlorothiazide, hydrocodone, hydromorphone, hydroxy- chloroquine, hydroxyzine, ibuprofen, imipramine, indomethacin, ketoconazole, ketoprofen, ketorolac, labetalol, levorphanol, lidocaine, loratadine, lorazepam, lovastatin, loxapine, mazin- dol, mefenamic acid, meperidine, mephénytoin, mepivacaine, mesoridazine, metaproterenol, metformin, methadone, methdilazine, methocarbamol, methotrexate, methotrimeprazine, methoxamine, methyl dopa, methylphenidate, metoclopramide, metolazone, metoprolol, met- ronidazole, midazolam, moclobemide, morphine, nadolol, nalbuphine, naloxone, naphazoline, naproxen, nifedipine, nizatidine, norepinephrine, nortriptyline, oxazepam, oxycodone, oxymet- azoline, paroxetine, pemoline, pentazocine, pentobarbital, pentoxifylline, perphenazine, pheno- barbital, phenol, phenolphthalein, phentolamine, phenylbutazone, phenyltoloxamine, phenyt- oin, pimozide, pindolol, piroxicam, pramoxine, prazepam, prazosin, probenecid, procainamide, procaine, prochlorperazine, procyclidine, promazine, promethazine, propafenone, propanthe- line, propiomazine, propofol, propranolol, protriptyline, quazepam, quinidine, quinine, race- methorphan, ranitidine, remoxipride, risperidone, salicylic acid, scopolamine, secobarbital, ser- traline, sotalol, spironolactone, sulfapyrazone, sulindac, temazepam, terbutaline, terfenadine, tetracaine, theophylline, thiethylperazine, thiopental, thioridazine, thiothixene, timolol, tocin- ide, tolbutamide, tolmetin, trazodone, triamterene, triazolam, trifluoperazine, triflupromazine, trimeprazine, trimethoprim, trimipramine, verapamil, warfarin, xylometazoline, yohimbine, zopiclone

**KEY WORDS**

details of plasma extraction

**REFERENCE**

Koves, E.M. Use of high-performance liquid chromatography-diode array detection in forensic toxicology, *J.Chromatogr.A*, **1995**, 692, 103–119.

# Phenmetrazine

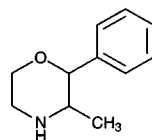
**Molecular formula:** C<sub>11</sub>H<sub>15</sub>NO

**Molecular weight:** 177.25

**CAS Registry No.:** 134-49-6, 1707-14-8 (HCl)

**Merck Index:** 7385

**Lednicer No.:** 1 260

**SAMPLE**

**Matrix:** blood

**Sample preparation:** Buffer 1 mL plasma to 9.1 with 50 mM Tris buffer, extract with diethyl ether. Extract the diethyl ether layer with 10 mM phosphoric acid, wash the aqueous layer with n-hexane, inject an aliquot.

**HPLC VARIABLES**

**Guard column:** CO:PELL:ODS

**Column:** 110 × 4.6 5 μm Partisphere C8 (Whatman)

**Mobile phase:** MeCN:10 mM pH 2.3 phosphate buffer 64:36

**Flow rate:** 0.45

**Detector:** UV 210

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**CHROMATOGRAM**

**Internal standard:** phenmetrazine

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**OTHER SUBSTANCES**

**Extracted:** reboxetine

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**KEY WORDS**

plasma; phenmetrazine is IS

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**REFERENCE**

Edwards,D.M.F.; Pellizzoni,C.; Breuel,H.P.; Berardi,A.; Castelli,M.G.; Frigerio,E.; Poggesi,I.; Rocchetti,M.; Dubini,A.; Strolin Benedetti,M. Pharmacokinetics of reboxetine in healthy volunteers. Single oral doses, linearity and plasma protein binding, *Biopharm.Drug Dispos.*, **1995**, 16, 443–460.

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**SAMPLE**

**Matrix:** bulk

**Sample preparation:** Mix a 1 mg/mL solution in 1 M sodium carbonate with 2 mL 5 mg/mL 8-quinolinesulfonyl chloride in acetone, heat at 65° for 20 min, cool, extract twice with 30 mL portions of chloroform. Combine the extracts and dry them over anhydrous magnesium sulfate, evaporate to dryness under a stream of air, reconstitute, inject an aliquot.

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**HPLC VARIABLES**

**Guard column:** 70 × 2.1 Co:Pell ODS

**Column:** 300 × 3.9 µBondapak C18

**Mobile phase:** MeCN:water:acetic acid 40:59:1

**Flow rate:** 1.5

**Detector:** UV 254, UV 280

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**CHROMATOGRAM**

**Retention time:** 16

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**OTHER SUBSTANCES**

**Simultaneous:** amphetamine, ephedrine, methamphetamine, phentermine, phenylpropanolamine, pseudoephedrine

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**KEY WORDS**

derivatization

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**REFERENCE**

Noggle,F.T., Jr.; Clark,C.R. Liquid chromatographic determination of primary and secondary amines as 8-quinolinesulfonyl chloride derivatives, *J.Assoc.Off.Anal.Chem.*, **1984**, 67, 687–691.

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**SAMPLE**

**Matrix:** solutions

**Sample preparation:** Prepare a 10 µg/mL solution in MeOH, inject a 20 µL aliquot.

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**HPLC VARIABLES**

**Column:** 125 × 4.9 Spherisorb S5W silica

**Mobile phase:** MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7

**Flow rate:** 2

**Injection volume:** 20

**Detector:** E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

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**CHROMATOGRAM**

**Retention time:** 2.4

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## OTHER SUBSTANCES

**Also analyzed:** acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzocetamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazepine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipiprone, diprenorphine, dipyrindamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothiopyndyl, isoxsuprine, ketanserine, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamylamine, meclorphenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylergonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, pimindine, pimozide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenylidamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranlycypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripeleminamine, triprolidine, tryptamine, verapamil, xylometazoline

## REFERENCE

Jane, I.; McKinnon, A.; Flanagan, R. J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J. Chromatogr.*, **1985**, *323*, 191-225.

## SAMPLE

**Matrix:** urine

**Sample preparation:** 500  $\mu$ L Urine + N-ethylordiazepam + chlorpheniramine + 100  $\mu$ L buffer, centrifuge at 11000 g for 30 s, inject a 500  $\mu$ L aliquot onto column A with mobile phase A, after 0.6 min backflush column A with mobile phase A to waste for 1.6 min, elute column A with 250  $\mu$ L mobile phase B, with 200  $\mu$ L mobile phase C, and with 1.15 mL mobile phase D. Elute column A to waste until drugs start to emerge then elute onto column B. Elute column B to waste until drugs started to emerge, then elute onto column C. When all the drugs have emerged from column B remove it from the circuit, elute column C with mobile phase D, monitor the effluent from column C. Flush column A with 7 mL mobile phase E, with mobile phase D, and mobile phase A. Flush column B with 5 mL mobile phase E then with mobile phase D. (Buffer was 6 M ammonium acetate adjusted to pH 8.0 with 2 M KOH.)

## HPLC VARIABLES

**Column:** A 10  $\times$  2.1 12-20  $\mu$ m PRP-1 spherical poly(styrene-divinylbenzene) (Hamilton); B 10  $\times$  3.2 11  $\mu$ m Aminex A-28 (Bio-Rad); C 25  $\times$  3.2 5  $\mu$ m C8 (Phenomenex) + 150  $\times$  4.6 5  $\mu$ m silica (Macherey-Nagel)

**Mobile phase:** A 0.1% pH 8.0 potassium borate buffer; B 6 mM  $\text{KH}_2\text{PO}_4$ , containing 5 mM tetramethylammonium hydroxide, and 2 mM dimethyloctylamine, pH adjusted to 6.50 with phosphoric acid; C MeCN:buffer 40:60 (Buffer was 6 mM  $\text{KH}_2\text{PO}_4$ , containing 5 mM tetramethylammonium hydroxide, and 2 mM dimethyloctylamine, pH adjusted to 6.50 with phosphoric acid.);

D MeCN:buffer 33:67 (Buffer was 6 mM  $\text{KH}_2\text{PO}_4$  containing 5 mM tetramethylammonium hydroxide, and 2 mM dimethyloctylamine, pH adjusted to 6.50 with phosphoric acid.); E MeCN:buffer 70:30 (Buffer was 6 mM  $\text{KH}_2\text{PO}_4$  containing 5 mM tetramethylammonium hydroxide, and 2 mM dimethyloctylamine, pH adjusted to 6.50 with phosphoric acid.)

**Column temperature:** ambient (column A), 40 (columns B and C)

**Flow rate:** A 5; B-E 1

**Injection volume:** 500

**Detector:** UV 210, UV 235

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#### CHROMATOGRAM

**Retention time:**  $k'$  2.7

**Internal standard:** N-ethylnordiazepam ( $k'$  2.1), chlorpheniramine ( $k'$  5.9)

**Limit of detection:** 300 ng/mL

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#### OTHER SUBSTANCES

**Extracted:** methamphetamine, desipramine, nortriptyline, diphenhydramine, methadone, imipramine, flurazepam, amitriptyline, morphine, codeine, hydromorphone, hydrocodone, caffeine, cotinine, benzoylecgonine, secobarbital, oxazepam, phenobarbital, nordiazepam, diazepam, phenylpropanolamine

**Interfering:** phentermine, amphetamine, lidocaine, ephedrine, pentazocine

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#### KEY WORDS

column-switching

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#### REFERENCE

Binder,S.R.; Regalia,M.; Biaggi-McEachern,M.; Mazhar,M. Automated liquid chromatographic analysis of drugs in urine by on-line sample cleanup and isocratic multi-column separation, *J.Chromatogr.*, **1989**, 473, 325–341.

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## Phenobarbital

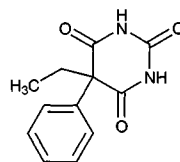
**Molecular formula:**  $\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}_3$

**Molecular weight:** 232.24

**CAS Registry No.:** 50-06-6, 57-30-7 (sodium salt)

**Merck Index:** 7386

**Lednicer No.:** 1 268



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#### SAMPLE

**Matrix:** blood

**Sample preparation:** Mix 500  $\mu\text{L}$  plasma with 500  $\mu\text{L}$  MeCN and 2  $\mu\text{g}$  IS for 30 s, centrifuge at 2700 g for 5 min, inject an aliquot of the supernatant.

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#### HPLC VARIABLES

**Column:**  $150 \times 4.6$  5  $\mu\text{m}$  Ultrasphere C18

**Mobile phase:** MeCN:MeOH:10 mM pH 7.4 phosphate buffer 15:35:50

**Column temperature:** 25

**Flow rate:** 1

**Detector:** UV 219

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#### CHROMATOGRAM

**Internal standard:** 2-hydroxy-2-ethyl-2-phenylacetamide

**Limit of detection:** 50 ng/mL

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#### OTHER SUBSTANCES

**Extracted:** carbamazepine, clonazepam, ethosuximide, D,L-2-hydroxy-2-ethyl-2-phenylpropionamide (HEPP), phenytoin, primidone